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Results of Routine Strip Effluent Hold Tank, Decontaminated Salt Solution Hold Tank, Caustic Wash Tank and Caustic Storage Tank Samples from Modular Caustic-Side Solvent Extraction Unit during Macrobatch 6 Operations

T. B. Peters

October 2013

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EXECUTIVE SUMMARY

Strip Effluent Hold Tank (SEHT), Decontaminated Salt Solution Hold Tank (DSSHT), Caustic Wash Tank (CWT) and Caustic Storage Tank (CST) samples from several of the "microbatches" of Integrated Salt Disposition Project (ISDP) Salt Batch ("Macrobatch") 6 have been analyzed for ^{238}Pu, ^{90}Sr, ^{137}Cs, and by Inductively Coupled Plasma Emission Spectroscopy (ICPES).

The results from the current microbatch samples are similar to those from comparable samples in Macrobatch 5.

From a bulk chemical point of view, the ICPES results do not vary considerably between this and the previous macrobatch.

The titanium results in the DSSHT samples continue to indicate the presence of Ti, when the feed material does not have detectable levels. This most likely indicates that leaching of Ti from MST in ARP continues to occur.

Both the CST and CWT samples indicate that the target Free OH value of 0.03 has been surpassed. While at this time there is no indication that this has caused an operational problem, the CST should be adjusted into specification.

The ^{137}Cs results from the SRNL as well as F/H lab data indicate a potential decline in cesium decontamination factor. Further samples will be carefully monitored to investigate this.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CF	Concentration Factor
CST	Caustic Storage Tank
CWT	Caustic Wash Tank
DF	Decontamination Factor
DSS	Decontaminated Salt Solution
DSSHT	Decontaminated Salt Solution Hold Tank
ICPES	inductively-coupled plasma emission spectroscopy
MCU	Modular Caustic-Side Solvent Extraction Unit
MST	monosodium titanate
SE	Strip Effluent
SEHT	Strip Effluent Hold Tank
SHT	Solvent Hold Tank
SRNL	Savannah River National Laboratory

1.0 Introduction

During operation of the ISDP, quantities of salt waste are processed through the Actinide Removal Process (ARP) and MCU in batches of ~3800 gallons. Monosodium titanate (MST) is used in ARP to adsorb actinides and strontium from the salt waste and the waste slurry is then filtered prior to sending the clarified salt solution to MCU. The MCU uses solvent extraction technology to extract cesium from salt waste and concentrate cesium in an acidic aqueous stream (Strip Effluent – SE), leaving a decontaminated caustic salt aqueous stream (Decontaminated Salt Solution – DSS). Sampling occurs in the Decontaminated Salt Solution Hold Tank (DSSHT) and Strip Effluent Hold Tank (SEHT) in the MCU process. The MCU sample plan[i] requires that batches be sampled and analyzed for plutonium and strontium content by Savannah River National Lab (SRNL) to determine MST effectiveness. The cesium measurement is used to monitor cesium removal effectiveness and the inductively coupled plasma emission spectroscopy (ICPES) is used to monitor inorganic carryover.

A previous report provided the results of several sets of sample results from Macrobatch 5 operations.[ii] Since that report, SRNL received subsequent SEHT, DSSHT, CWT and CST samples from Macrobatch 6 (1/2013 to 5/2013).

2.0 Experimental Procedure

The samples were contained in 10-mL P-nut vials. SEHT samples were delivered in doorstops for shielding purposes, while the CWT, CST and DSSHT samples were delivered in thief holders. Samples were removed from the holders. The CWT, CST and DSSHT samples were sent for analysis without dilution or filtration. SEHT samples were sent with dilution but without filtration.

2.1 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.

3.0 Results and Discussion

The radiochemical results from the DSSHT and SEHT analyses are listed in Table 1. Under normal operations, there is only one transfer from Tank 21H to Tank 49H in each macrobatch. In order to improve operational efficiency, there have been a total of 4 transfers to Tank 49H. This in turn generates 4 slightly different batches of material; labeled 6-A, 6-B, 6-C, and 6-D. The ^{238}Pu, ^{90}Sr and ^{137}Cs content in each batch varies by ~6% or less. Therefore, entries in the "Source Material" column are averages of all four batches.[iii,iv,v]

Table 1. Radiochemical Results for the DSSHT and SEHT Results

Sample ID	Sample Date	^{238}Pu (dpm/mL)	^{90}Sr (dpm/mL)	^{137}Cs (dpm/mL)
DSSHT Samples				
MCU-13-158	1/28/2013	3.00E+03 (5.39%)	4.30E+03 (10.5%)	7.31E+05 (5.00%)
MCU-13-339	3/4/2013	6.97E+02 (5.08%)	2.85E+03 (10.3%)	7.43E+05 (5.00%)
MCU-13-474	3/26/2013	9.17E+02 (5.68%)	4.09E+03 (10.2%)	8.15E+05 (5.00%)
MCU-13-613	4/22/2013	1.94E+03 (5.79%)	2.13E+03 (10.9%)	9.70E+05 (5.00%)
MCU-13-809	5/14/2013	1.18E+03 (6.98%)	3.05E+03 (11.6%)	1.06E+06 (5.00%)
SEHT Samples				
MCU-13-162	1/30/2013	<6.24E+01	9.41E+03 (10.5%)	1.85E+09 (5.00%)
MCU-13-343	3/5/2013	<3.68E+01	3.41E+03 (11.9%)	2.04E+09 (5.00%)
MCU-13-475	3/26/2013	<8.13E+00	<2.60E+02	1.68E+09 (5.00%)
MCU-13-617	4/22/2013	<7.58E+00	1.71E+03 (12.2%)	1.78E+09 (5.00%)
MCU-13-805	5/14/2013	1.09E+02 (15.5%)	5.32E+03 (13.5%)	1.75E+09 (5.00%)
Source Material (average of 6-A, 6-B, 6-C and 6-D)		2.91E+04	4.14E+05	1.30E+08

While we do not have many data points for this Macrobatch, what we do have shows a similar pattern to the overall behavior from Macrobatch 5 operations. Table 2 lists the average Decontamination Factor (DF) values for ^{238}Pu, ^{90}Sr and ^{137}Cs for both Macrobatch 5 and 6.[Π] The values in parentheses are the % relative standard deviation.

Table 2. Average DF Values from Macrobatch 5 and 6

Isotope	Average Macrobatch 6 DF	Average Macrobatch 5 DF
^{238}Pu	24.4 (52.9%)	35.6 (44.4%)
^{90}Sr	134 (29.1%)	184 (41.7%)
^{137}Cs	154 (16.0%)	289 (33.1%)

The purpose in comparing the two macrobatches is to establish that the average decontamination of these three isotopes is approximately the same. Given the differences in the feed and in operating conditions, some variation in the DF values is expected. For example, the difference in the DF values for ^{137}Cs should not be taken as Macrobatch 6 necessarily being much less efficient in cesium removal. The high %RSD also makes it problematic to make direct comparisons. Furthermore, during Macrobatch 5 operations before October 2012, ARP was using a larger MST strike and time, which biases part of the DF values for Pu and Sr higher for Macrobatch 5.

Figure 1 shows the graph of the ^{238}Pu results in the DSSHT for all of the Macrobatch 6 DSSHT samples. Figure 2 shows the same for ^{90}Sr. Figure 3 shows the similar ^{137}Cs

[Π] Recall that DF is defined as the feed value divided by the DSSHT sample value.

data, but also includes the SEHT sample results. Figure 4 shows the concentration factor (CF) over time.

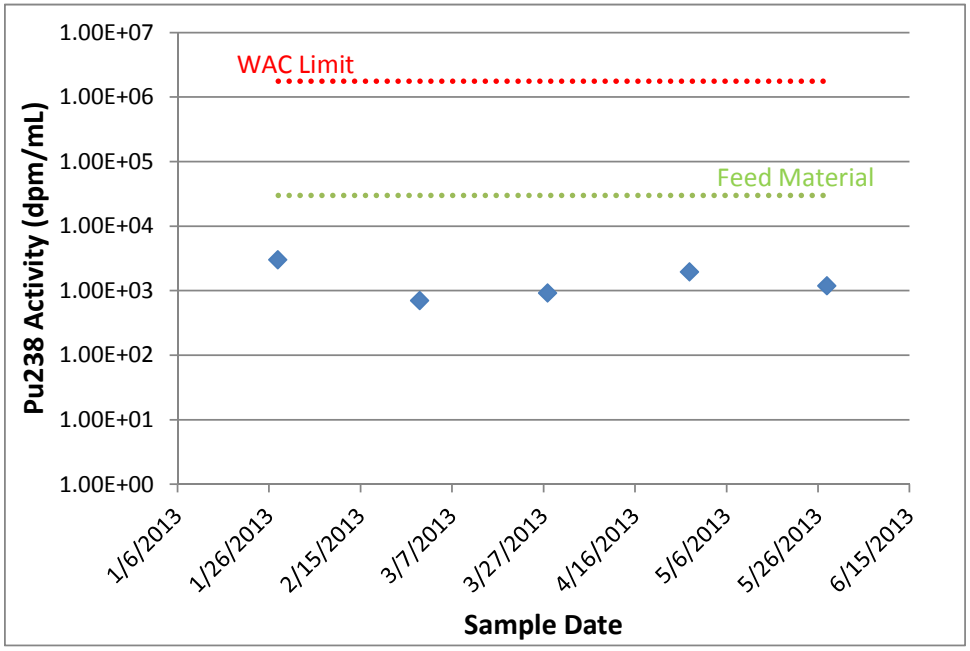

Figure 1. ^{238}Pu Data for Macrobatch 6 DSSHT Samples

While the graph of the Pu and Sr data can show the overall trend, it is also important to consider the flow rates as recorded in the facility, as well as the periodicity of the removal of the MST filter cake.

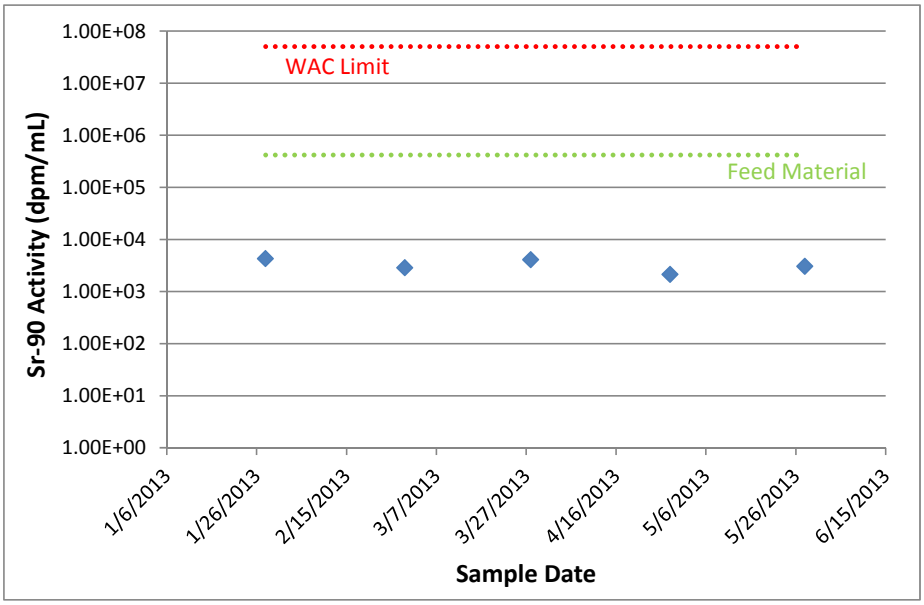

Figure 2. ^{90}Sr Data for Macrobatch 6 DSSHT Samples

For the ^{137}Cs results, both the DSSHT and SEHT results are shown. See Figure 3. The DSSHT samples are all well below the WAC limit, and the SEHT samples give an average concentration factor of 14.0 (3.90% RSD).[▽] See Figure 4.

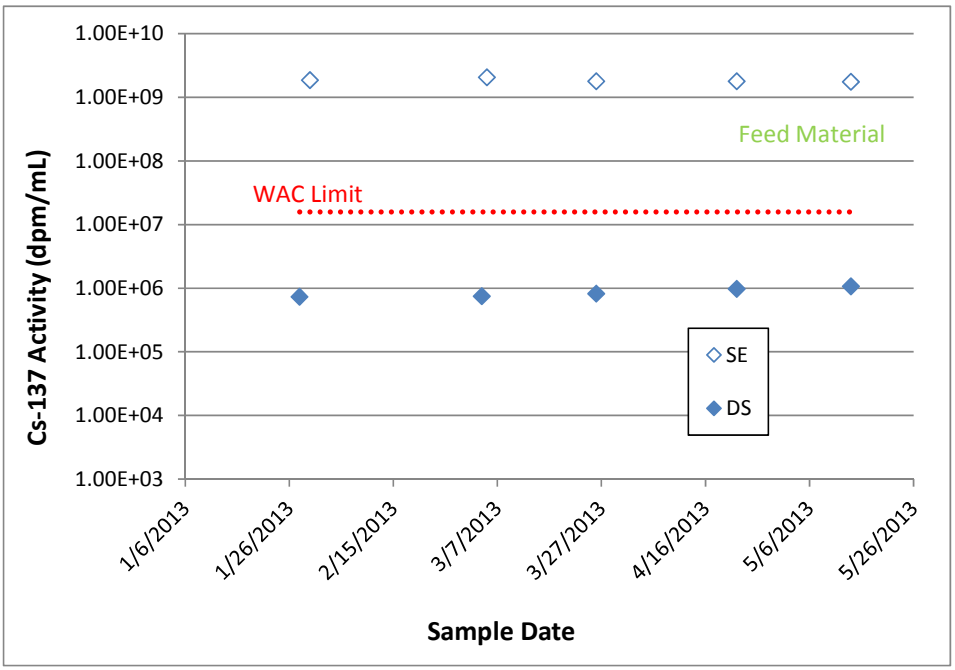

Figure 3. ^{137}Cs Data for Macrobatch 6 DSSHT and SEHT Samples

There appears to be a slight upward trend in the DSSHT values, which in turn drives the DF lower. When additional data becomes available, SRNL will evaluate whether or not this is a real trend caused by some process parameter.

The CF values for macrobatch 6 so far are very close to that from Macrobatch 5, which averaged 12.9 (10.9% RSD).

[▽] The concentration factor (CF) is defined as the SEHT value divided by the feed value.

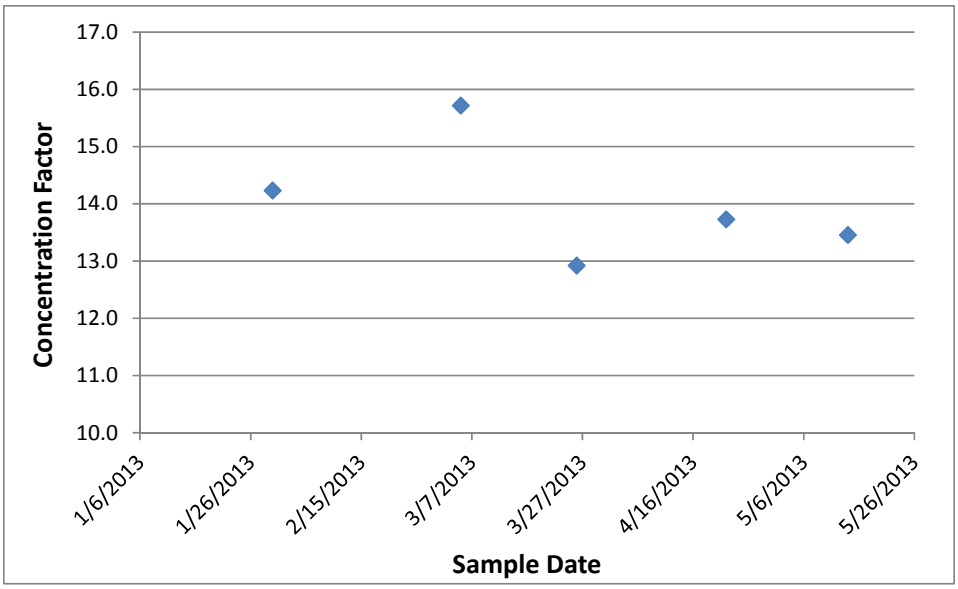

Figure 4. Concentration Factor For Macrobatch 6 Samples

The CF value from the second data point in the series is slightly higher than the other samples which are due to the slightly higher SE results.

During the sample period, F/H Laboratory was analyzing DSSHT and SEHT samples for ^{137}Cs content. Figure 5 shows the comparison of results between SRNL and F/H Lab.

On average, SRNL and F/H Lab results were ~5% different for the DSSHT samples, and ~16% different for the SE samples.

The same slight increase in DSSHT values over time is also noted in the comparative F/H lab data (Figure 5) as well as in the larger set of DSSHT sample results from F/H lab.

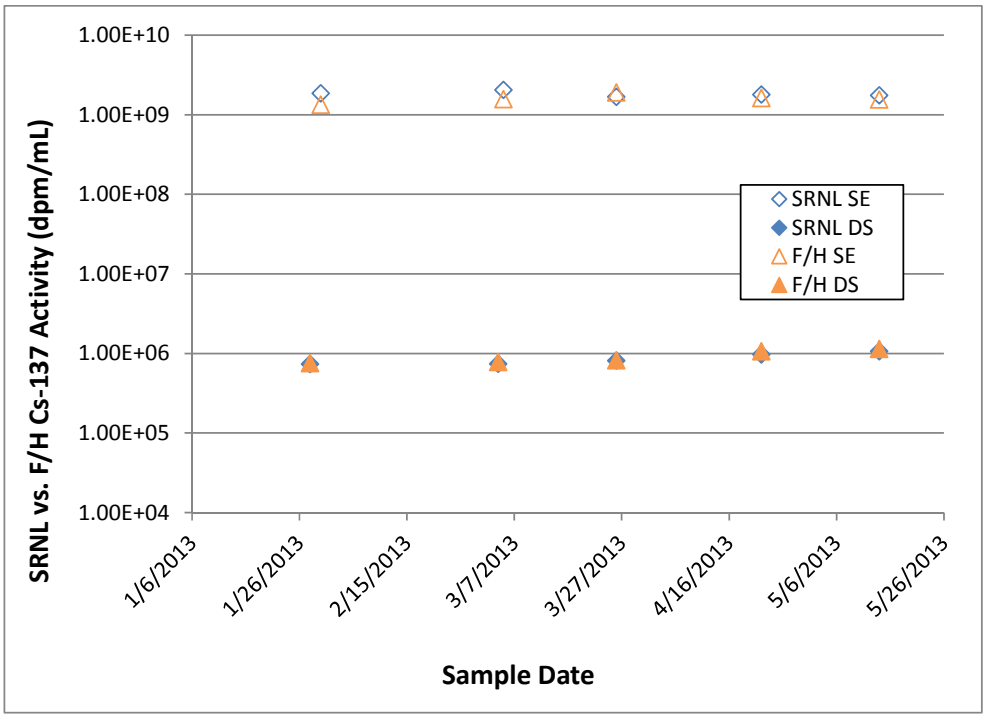

Figure 5. Comparison of SRNL and F/H Lab ^{137}Cs Sample Results

The meaningful (present in non-trace quantities) ICPES results for the DSSHT samples are listed in Table 3, and the meaningful ICPES results for the SEHT samples are listed in Table 4. As there are four different sub-batches (6-A, 6-B, 6-C, 6-D), the average dilution, across the four sub-batches, is recorded in the right most column.[ƒ] As titanium is introduced to the system via MST leaching at ARP, there is no entry for this element. Note that material from Tank 49H undergoes a ~17 vol % dilution from ARP and MCU.[ϒ] Therefore, direct comparisons between the source material and the DSSHT sample results should take this into account. We note that a comparison of several of the more concentrated analytes (Al, B, Cr, K, Na, P, and S) gives an average dilution to ~86%, indicating that additional sources of dilution have largely been avoided. The initial DSSHT samples (MCU-13-158) give consistently lower analyte concentrations than the later samples, and this may be an indication that this first sample of Salt batch 6 had some residual water flush or other dilution.

[ƒ] While the pump suction in Tank 49 is around 40 inches from the tank bottom, transfers into Tank 49 enter the tank through a short un-submerged downcomer. Initial feed composition is more like the end of Salt batch 5. As transfers progress 6-A, 6-B, 6-C, 6-D, and the level in Tank 49 is reduced due to MCU operation, feed composition will more become more like salt batch 6 Tank 21H material.

[ϒ] Each 3715 gallon batch of Tank 49H material is mixed with 105 gallons of MST slurry, and is then combined with 256 gallons of scrub acid and 256 gallons of caustic wash. This dilutes each 3715 gallons to 4332 gallons, or ~17 vol % increase in volume.

The titanium results in the DSSHT samples are notable. In all cases, we have greater than detectable levels of Ti in the samples, where there is less-than detectable amounts in the feed material. This is important, as the only possible source of Ti is from the MST used at ARP. In fact, SRNL has found evidence of Ti-containing solids in the DSSHT coalescer and pre-filters. Testing at SRNL has shown that Ti can be leached from MST solids in a caustic environment; this is suspected as a contributor to the Ti component in the MCU samples.[vi]

Table 3. ICPES Results for the DSSHT Samples (mg/L)

	MCU-13-158	MCU-13-339	MCU-13-474	MCU-13-613	MCU-13-809	Average Dilution
Sample date	1/28/13	3/4/13	3/26/13	4/22/13	5/14/13	
Al	3310	4020	4060	4660	4400	69.7%
B	38.1	40.1	41.6	45.0	43.9	103%
Cr	31.5	34.6	34.6	40.3	37.4	84.3%
K	225	241	258	306	289	73.9%
Na	101000	116000	118000	131000	124000	80.5%
P	127	136	142	158	158	99.0%
S	1790	2310	2020	2230	2220	92.8%
Ti	2.18	2.97	2.71	4.2	3.1	NA
Zn	4.65	4.18	3.65	7.27	4.99	82.3%

The analytical uncertainty for the ICPES samples is 10%.

Table 4 .ICPES Results for the SEHT Samples (mg/L)

	MCU-13-162	MCU-13-343	MCU-13-475	MCU-13-617	MCU-13-805
Sample Date	1/30/13	3/5/13	3/26/13	4/22/13	5/14/13
Al	8.31	8.70	<1.12	<1.20	<1.19
B	<0.345	<0.359	<0.335	<0.360	<0.356
Ba	0.210	0.176	<0.148	<0.159	<0.157
Ca	5.93	6.22	1.12	0.76	0.65
Cr	<0.642	<0.667	<0.623	<0.669	<0.662
Fe	12.6	12.5	<0.34	<0.37	0.70
K	<6.85	9.96	<10.1	<10.8	<7.07
Mg	0.541	0.556	0.411	0.153	0.102
Na	66.5	82.9	70.4	60.6	27.8
P	<4.80	<5.00	<4.67	<5.01	<4.96
S	<173	<179	<168	<180	<178
Ti	<0.167	<0.173	<0.162	<0.174	<0.172
Zn	14.2	14.6	0.82	1.10	<0.48

The analytical uncertainty for the ICPES samples is 10%.

The SEHT samples follow the general trends observed for the previous sample results – most analytes are below detection limits.[ii] There is a notable decline in many of the results between the February (MCU-13-343) and March (MCU-13-475) samples (Al, Ca, Fe). Taken together, these results might indicate that MCU had some degree of high carryover in macrobatch 5, which is declining in macrobatch 6. Alternatively, the moderate levels of these elements present in the initial samples may have declined if they happened to be tramp elements present in an upstream cold feed that is no longer being added as often.

3.1 Caustic Storage (CST) Sample

A single sample (MCU-13-648) from the Caustic Storage Tank was sent to SRNL for analysis. Samples of this material were analyzed without dilution. The results are reported in Table 5.

Table 5. Results for the CST Sample

Analyte	Result
Suspended Solids	<0.01 wt%
Total Inorganic Carbon	7.24 (μgC/mL)
Total Organic Carbon	<4 (μgC/mL)
Na	966 mg/L
Free OH	0.037 M
Fluoride	<10
Formate	<10
Chloride	16
Nitrite	<10
Bromide	<10
Nitrate	<10
Phosphate	<10
Sulfate	<10
Oxalate	<10

The uncertainty on each analysis is 10%.

The solution is supposed to be a 0.03 M NaOH solution, which gives a target sodium value of 690 mg/L. Both the Free OH and sodium results are higher than expected. The other results do not indicate other issues.

3.2 Caustic Waste Tank (CWT) Samples

Five samples from the CWT (MCU-13-627, -628, -629, -630, -631) were delivered to SRNL for analysis. These samples were composited and sent to AD for analysis. The results are reported in Table 6.

Table 6. Results for the CWT Sample Composite

Analyte	Result
pH	12.5
^{137}Cs	1.52E+04 dpm/mL
Suspended Solids	0.1 wt%
Total Inorganic Carbon	22.6 (µgC/mL)
Total Organic Carbon	40.4 (µgC/mL)
Na	1300 mg/L
Free OH	0.0444 M
TOA (SVOA)	<10 mg/L
Isopar L (SVOA)	<33 mg/L
Modifier (SVOA)	17 mg/L
Modifier (HPLC)	26 mg/L
Fluoride	<10
Formate	<10
Chloride	10
Nitrite	<10
Bromide	<10
Nitrate	26
Phosphate	<10
Sulfate	<10
Oxalate	<10

The analytical uncertainty is 20% for the Semi Volatile Organic Analysis (SVOA) samples, 5% for the ^{137}Cs measurement and 10% for the others.

The results are largely as expected although the Free OH and sodium levels are above expectations. This is not unexpected given that the caustic feed from the CST (see section 3.1) is also above expectations for those analytes. The ^{137}Cs value is within the range of previously reported values.[vii] The measurements for the Modifier are higher than a previous report.[viii] The increase in the Modifier in the CWT samples may be due to the increase in caustic concentration (0.01 to 0.03 M) that took place after the previous Modifier measurements.[π] However, this is from a single data point and additional data will be looked at in future samples. In the current sample, the Modifier, averaging 21.5 mg/L between the two measurements, is low enough such that this is probably near the solubility limit. However, if future CWT samples show this level of Modifier being present, consideration should be given to whether or not this is an indication of non-trivial Modifier depletion. At this time, there has been no need to add Modifier to correct a perceived decline in Modifier concentration.

[π] The Cs-7SB Modifier has an alcohol functionality which is subject to deprotonation. Increased Free Hydroxide in the caustic wash would generate higher concentrations of the more aqueous soluble deprotonated form of the Modifier.

For the SVOA and HPLC measurements, no other analytes were detected, including *sec*-butyl phenol or TOA, to a detection limit of 10 mg/L. A later CWT sample was delivered to SRNL for analysis; MCU-13-810. This sample was analyzed and the results are reported in Table 7.

Table 7. Results for the MCU-13-810

Analyte	Result
pH	12.5
^{137}Cs	5.25E+03 dpm/mL (5.79%)
Na	833 mg/L (10%)

The value in parentheses is the analytical uncertainty. The analytical uncertainty is typically <1% for density measurements and typically 0.5 pH units for the pH measurements.

Compared to the previous CWT sample composite, the ^{137}Cs decline is notable, but within previous experience.[vii] The sodium result is closer to the expected value of 690 mg/L.

3.3 Modeling the MCU System from Output Samples

Historically, the DS, SE, CWT and Solvent Hold Tank (SHT) samples arrive at divergent times, making it impossible to attempt to establish a ^{137}Cs activity balance at MCU.[∅] The May samples reported in this document, along with the SHT sample reported separately, [ix]are all from approximately the same time period, making direct comparisons valid. SRNL is in the process of developing a model that compares sample sets taken at the same time, in order to derive the activity balance and operating information. As further sample sets arrive, SRNL will be able to provide a more clear understanding of how the output samples reflect internal processing.

4.0 Conclusions

The results from the current microbatch samples are similar to that from comparable samples in Macrobatch 5. From a bulk chemical point of view, the ICPES results do not vary considerably between previous results and this macrobatch.

The Pu and Sr results show there are no process unsets at ARP as the DSSHT continues to show acceptable decontamination.

[∅] An activity balance is defined as the total activity in the output (DS, SE, CWT and SHT), divided by the total activity in the input (material from ARP) to MCU. Ideally this should be 100%.

The titanium results in the DSSHT samples continue to indicate the presence of Ti, when the feed material does not have detectable levels. This most likely indicates that leaching of Ti from MST has increased in ARP at the higher free hydroxide concentrations in the current feed.

Both the CST and CWT samples indicate that the target Free OH value of 0.03 has been surpassed. While at this time there is no indication that this has caused an operational problem, the CST should be adjusted into specification.

The ^{137}Cs results from the SRNL as well as F/H lab data indicate a potential decline in cesium decontamination factor. Further samples will be carefully monitored to investigate this.

5.0 Path Forward

SRNL recommends that in the future, when samples are delivered, the customer should make a note of how many MST additions have been made before the sample was pulled. This will enable SRNL to more accurately understand the effects of MST on the Pu and Sr results.

A future sample from the CST is warranted given that it is out of specification.

SRNL recommends analyzing two more concurrent sets of the DS, SE, and CWT samples for Modifier and TOA content in order to establish whether or not non-trivial depletion is occurring.

Appendix A Analyte Concentrations in Each Feed Batch

During processing of Salt Batch 6 to date, there have been 3 additional transfers of Tank 21H (beyond the original one for Salt Batch 6) to Tank 49H. This has created subtle differences in the feed material to ARP and MCU. In order to compare the feeds to the ICPES data, Table 8 shows selected ICPES results for each of the 4 sub-batches to date (6-A,[iii] 6-B,[iii] 6-C,[iv] and 6-D[v]).

Table 8. Selected ICPES Results for the 4 Sub-Batches (mg/L)

Analyte	6-A	6-B	6-C	6-D
Al	6507	6372	6264	6183
B	38.7	39.5	40.1	40.5
Ba	0.654	0.692	0.719	0.739
Ca	<0.91	<0.91	0.98	<0.91
Cr	42.4	42.8	43.1	43.3
Fe	4.74	4.51	4.34	4.22
K	359	371	379	385
Mg	<0.1	0.155	<0.1	<0.1
Na	151110	151340	151340	151340
P	146	144	141	140
S	2317	2298	2282	2269
Ti	2.18	2.97	2.71	NA
Zn	4.6	4.66	4.7	4.73

The analytical uncertainty for the ICPES samples is 10%
NA = This value was not calculated

6.0 References

[i] M. W. Geeting, "Interim Salt Disposition Project (ISDP) Sample Plan", U-ESR-H-00068, Revision 5, April 28, 2009.

[ii] Peters, T. B., Fondeur, F. F., "Results of Routine Strip Effluent Hold Tank and Decontaminated Salt Solution Hold Tank Samples from Modular Caustic-Side Solvent Extraction Unit during Macrobatch 5 Operations" SRNL-STI-2013-00194, April 2013.

[iii] S. E. Campbell, "Blend Evaluation for Tank 49 Feed for ISDP Salt Batch 6", X-ESR-H-00468, February 21, 2013.

[iv] S. E. Campbell, "Blend Evaluation for Tank 49 Feed for ISDP Salt Batch 6-C", X-ESR-H-00489, April 13, 2013.

[v] S. E. Campbell, "Blend Evaluation for Tank 49 Feed for ISDP Salt Batch 6-D", X-ESR-H-00501, April 23, 2013.

[vi] K. M. L. Talyor-Pashow, F. F. Fondeur, S. D. Fink, "Leaching of Titanium from Monosodium Titanate (MST) and Modified MST (mMST)", SRNL-STI-2012-00237, August 2012.

[vii] T. B. Peters, S. D. Fink, "Results of Routine Strip Effluent Hold Tank, Decontaminated Salt Solution Hold Tank, and Caustic Wash Tank Samples from Modular Caustic-Side Solvent Extraction Unit during Macrobatch 4 Operations", SRNL-STI-2013-00430, October 2012.

[viii] F. F. Fondeur, S. D. Fink, "Characterization and Evaluation of Caustic Wash Tank and Solvent Hold Tank Samples from MCU from August to September 2011 (SHT 221-226, SHT 314-319, SHT 1035-1040, and CWT 817-904)", SRNL-STI-2012-00324, August 2012.

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